PRODUCTION OF GREASE FROM SPENT LUBRICANT

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I declare that this thesis entitled "*Production of Grease from Spent Lubricant*" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Date	: 20 April 2009

Special Dedication of This Grateful Feeling to My…

Beloved parent;

Mr. Awang Ismail b Awang Rambli & Mrs. Dara bt Sahari

Loving brother and sisters;

Awang Morshidi and Dayang Nur Fitrah

Understanding and helpful friends;

For Their Love, Support and Best Wishes.

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ABSTRACT

This study was undertaken to examine the potential of spent lubricant as a medium to produce grease. Grease is a semi-solid colloidal dispersion of a thickener in a liquid lubricant matrix. Spent lubricant oils have significant potential as a base fluid and a substitute for mineral oil in grease formulations. The advantages of using products based on lubricant oils are their physical and chemical properties are close to that common grease production. Besides, this research allowing as optimizing the oil waste usage and reduce the environmental problem towards waste disposal. This work focuses on the preparation methods, optimization of thickener components, and antioxidant additive for producing spent lube oil based grease. In greases, Li-fatty acid thickener having C_{12} to C_{18} chain lengths and different metal to fatty acid ratios were synthesized. The percentages of thickener were varied at 5%, 10%, 15% and 20%. The research also focuses on varying the mixing temperature during the saponification of grease. Four sets of reaction temperature were 150◦C, 170◦C, 200◦C, and 230◦C. Four types of grease properties or characteristic to be tested in this research were grease oxidation stability, penetration, dynamic viscosity, and dropping point. Results indicate that Li-thickener composition, fatty acid types and base oil content and reaction temperature significantly affect grease hardness, dynamic viscosity, oxidation, and dropping point. It has been observed that the physical and chemical properties of grease are largely based on the thickener fibre micro-structure.

ABSTRAK

Kajian ini dijalankan untuk mengkaji keupayaan atau potensi minyak hitam (pelincir) terpakai sebagai medium untuk menghasilkan gris. Gris merupakan bahan separa pejal yang mengandungi serakan bahan pemekat yang dipanggil thickener. Minyak hitam terpakai mempunyai potensi yang tersendiri sebagai minyak asas bagi mengantikan penggunaan minyak mineral komersil dalam pembuatan gris. Minyak hitam terpakai mempamerkan beberapa kelebihan yang tersendiri berasaskan fizikal dan kimianya yang menghampiri sifat-sifat gris. Kajian ini memberikan peluang untuk kita mengoptimumkan penggunaan bahan terpakai sekaligus mengurangkan masalah terhadap alam sekitar. Kajian memfokus kepada metod pembuatan gris, penggunaan optimum cecair pemekat dan penggunaan bahan tambahbaikan untuk menghasilkan gris berasaskan minyak hitam terpakai. Dengan ini, peratus cecair pemekat akan dipelbagaikan iaitu 5%, 10%, 15%, dan 20% daripada keseluruhan gris. Di samping itu, kajian in juga memfokuskan kepelbagaian penggunaan suhu ketika proses saponifikasi. Untuk itu, empat set suhu akan digunapakai iaitu 150°C, 170°C, 200°C, dan 230°C. Keputusan kajian menunjukkan beberapa impak penting terhadap cirri-ciri gris berdasarkan kaedah mempelbagaikan peratus cecair pemekat serta suhu untuk proses saponifikasi. Antara cirri-ciri gris yang dikaji ialah kekerasan gris, kelikatan gris, pengoksidaaan dan suhu pencairan gris. Pemerhatian menunjukkan sifat-sifat gris banyak dipengaruhi oleh cecair pemekat yang akan membentuk mikrostuktur gris tersebut

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CP	-	Centi Poise
$NaS_2CN(C_2H_5)_2$	-	Sodium Diethyldithiocarbamate
LiOH	-	Lithium Hydroxide
TFMO	-	Thin Film Micro Oxidation
◦C	-	Degree Celsius
◦F	-	Degree Fahrenheit

APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Grease, as it is looked upon today, is a relatively new science. In 1400 B.C., mutton or beef fat was sometimes mixed with lime to reduce the friction in chariot wheels. Under the correct conditions, heating lime and fat in oil will form grease. Modern greases, however, were not commercially available until more than 3,300 years later. The first grease produced in volume was calcium soap grease. Lithium, barium and calcium complex greases were introduced in the 1930s and 1940s. Aluminum complex greases followed in the early 1950s, but modern lithium complex greases did not enter the market until the early 1960s. (Sandra Cowan, 2007)

Conversely, the worldwide production reports higher percentages of hydrated calcium, conventional lithium and sodium soap grease. This could be due to a difference in equipment lubrication demands in various parts of the world. In general, high-speed or heavily loaded equipment can generate more heat, which creates an increased need for greases with higher dropping points. In addition, higher labor costs in North America factor into the need to extend relubrication intervals and therefore increase the need for grease that can function for longer periods of time. (NLGI Annual Grease Survey, 2005)

As industrial growth rapidly, the grease production industries become wider. More technologies being develop to produce a high-end quality of grease. Currently, many researchers have been carried out to search alternative sources for grease production due to increasing of crude oil price in the world market, thus affecting the price of base oil as main component in grease. Instead of using commercial oil as based, other potential sources which are more economic and abundantly available are soybean oil, synthetic oil and waste lubricant.

1.2 Problem of Statement

Crude oil or mineral oil is one of the most valuable compounds in the world. Crude oil products contribute major application within industries such as automotive, machinery, building and medicine. Lubricant oil is one of the application products from crude oil. In machinery and automotive industries, the lubricant oil is applied onto the surface of moving part such as rotor and ball bearing. It also acts as a surface seal to inhibit corrosion (Cavengros, J. *et al.,* 2002). Lubricant oil has several characteristics which contribute for its long life spent inside the machine. But, after certain years, the lubricant oil will loss it characteristic and become vulnerable. This is due to contaminant or metal waste trap inside the lubricant oil. As lube oil become harder, it will scratch the performance of the machine and need to replace. Thus, this lubricant is claim as waste lubricant. Several techniques are available to treat this waste lubricant so that it can be reused in other application.

From the research by Prakash K Ramdoss *et al.*, those wastes were not manage appropriately and it just disposes through land filling and this scenario become worst. As record recently in 2002 worldwide, this number soars to about 5.3 billion gallons. Of the amount generated in the United States each year, over 400 million gallons are largely not recovered and presumed to be disposed of improperly, creating significant environmental problems. Of the approximately 900 million gallons collected, only about 140 million are reprocessed (Lundberg J. e*t al.*, 2003). Recently, vast researches of grease production claim to use vegetable oil such as waste cooking oil or soybean oil to prepare the grease (Brajendra K. Sharma *et al.*, 2006). However, there are no specific researches on the grease production that use spent lubricant as the base oil component of the grease. It is due to the high viscosity of the lubricant itself, thus it is hard to handle. Since the lubricant base is come from mineral oil, it is not impossible to make grease from spent lubricant with a high quality and economically viable.

1.3 Objectives

To produce high quality grease from spent lubricant and comparing the quality with commercial grease.

1.4 Scope of Research

In attempt to understand the characteristic of lubricating grease, there are some influence factors that must be study to ensure the grease meet the quality of industrial standard and long life performance. In order to achieve the objectives, several scopes has been identified.

i. To investigate the effect of thickener percentage composition towards the oxidation stability of the grease. Thickener compositions were varied 5%, 10%, 15%, and 20% w/w.
ii. To study the effect of thickener towards consistency of grease
iii. To study the effect of thickener towards apparent viscosity of grease.
iv. To investigate the effect of thickener towards grease dropping point.
v. To study the effect of mixing temperature towards the formation of grease. Mixing temperatures were varied 150°C, 170°C, 200°C, and 230°C.

CHAPTER 2

LITERATURE REVIEW

2.1.1 Grease Anatomy

Grease is a semi fluid combination of a fluid lubricant, a thickener, and additives. The fluid lubricant or the based oil that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil (Lundberg J *et al.,* 2003). The thickener gives grease its characteristic consistency and is sometimes thought of as a "three-dimensional fibrous network" or "sponge" that holds the oil in place (Crone I. *et al.,* 2003). Common thickeners are soaps and organic or inorganic nonsoap thickeners. The majority of greases on the market are composed of mineral oil blended with a soap thickener. Additives enhance performance and protect the grease and lubricated surfaces. Figure 2.1 shows the general composition of base oil, thickener and additive to produce grease. The composition can be manipulated to get different properties and function of grease. From the figure, it shows that the base oil can be divided into mineral oil or synthetic oil. Thickener or soap can be classified as simple soap, complex, and non soap. Moreover, the existence of additive can create new properties as well as enhance the performance of grease (Aihara C. *et al.,* 1995).

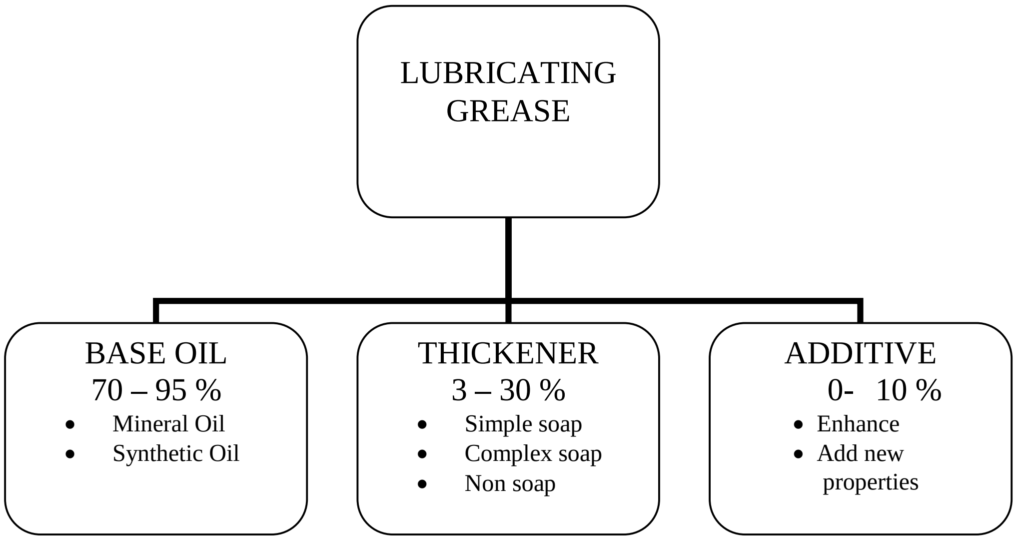

Figure 2.1 Grease Anatomies (After (Jeremy Wright, 2001))

2.2 Base Oil

Most grease produced today use mineral oil as their fluid components. These mineral oil-based greases typically provide satisfactory performance in most industrial applications. In this research spent lubricant is utilized as base oil. There are five specific categories of base oils. These categories define the type of base stock the oil is formulated from. The categories are as follows.

2.2.1 Group I - Solvent Freezing (M. A. Delgado *et al*., 2006)

Group 1 base oils are the least refined of all the groups. They are usually a mix of different hydrocarbon chains with little or no uniformity. While some automotive oils on the market use Group I stocks, it's generally used in less demanding applications.

2.2.2 Group II - Hydro processing and Refining (Mazuyer D. *et al.*, 2003)

Group II base oils are common in mineral based motor oils currently available on the market. It have fair to good performance in lubricating properties such as volatility, oxidative stability and flash/fire points. It gives fair performance in areas such as pour point, cold crank viscosity and extreme pressure wear.

2.2.3 Group – III Hydro processing and Refining (Chin –Hsian Kuo *et al.*, (1996)

Group III base oils are subjected to the highest level of mineral oil refining of the base oil groups. Although it is not chemically engineered, it offers good performance in a wide range of attributes as well as good molecular uniformity and stability. They are commonly mixed with additives and marketed as synthetic or semi-synthetic products. Group III base oils have become more common in America in the last decade.

2.2.4 **Group IV -Chemical Reactions** (Sevim Z. Erhan *et al.*, 2006)

Group IV base oils are chemically engineered synthetic base stocks. Polyalphaolefins (PAO's) are a common example of a synthetic base stock. Synthetics, when combined with additives, offer excellent performance over a wide range of lubricating properties. They have very stable chemical compositions and highly uniform molecular chains. Group IV base oils are becoming more common in synthetic and synthetic-blend products for automotive and industrial applications.

2.2.5 **Group V** (Giordin D. *et al.*, 2003)

Group V base oils are used mainly in the creation of oil additives. Esters and polyolesters are both common Group V base oils used in the formulation of oil additives. Group V oils are generally not used as base oils themselves, but add beneficial properties to other base oils.

Most grease is formulated using Group I and II mineral oil base stocks, which are appropriate for most applications. However, there are applications that might benefit from the use of synthetic base oil. Such applications include high or low operating temperatures, a wide ambient temperature range, or any application where extended relubrication intervals are desired.

2.3 Lubricant oil

Motor oil is a lubricant used in internal combustion engines. These include motor or road vehicles such as cars and motorcycles, heavier vehicles such as buses and commercial vehicles, non-road vehicles such as go-karts, snowmobiles, boats (fixed engine installations and outboards), lawn mowers, large agricultural and construction equipment, trains and aircraft, and static engines such as electrical generators. In engines there are parts which move very closely against each other causing friction which wastes otherwise useful power by converting the energy to heat. Contact between moving surfaces also wears away those parts, which could lead to lower efficiency and degradation of the motor. This increases fuel consumption and decreases power output and can, in extreme cases, lead to total engine failure (Cavengros, J. *et al.*, 2002).

Lubricating oil creates a separating film between surfaces of adjacent moving parts to minimize direct contact between them, decreasing friction, wear, and production of excessive heat, thus protecting the engine. Motor oil being a good conductor of heat, it is brought into contact with a hot surface, thereby absorbing some of the heat from said surface so the oil can then transfer the heat elsewhere, typically to the air or a heat sink of some variety.

Coating metal parts with oil keeps them from being exposed to oxygen, inhibiting oxidation at elevated operating temperatures preventing rust or corrosion. Corrosion inhibitors may also be added to the motor oil. Many motor oils also have detergent and dispersant additives to help keep the engine clean and minimize oil sludge build-up.

Rubbing of metal engine parts inevitably produces some microscopic metallic particles from the wearing of the surfaces. Such particles could circulate in the oil and grind against the moving parts, causing erosion and wear. Because particles inevitably build up in the oil, it is typically circulated through an oil filter to remove harmful particles. An oil pump, a vane or gear pump powered by the vehicle engine, pumps the oil throughout the engine, including the oil filter. Oil filters can be a full flow or bypass type (Cavengros, J. *et al.*, 2002).

In the crankcase of a vehicle engine, motor oil lubricates rotating or sliding surfaces between the crankshaft journal bearings (main bearings and big-end bearings), and rods connecting the pistons to the crankshaft. The oil collects in an oil pan, or sump at the bottom of the crankcase. In some small engines such as lawn mower engines, dippers on the bottoms of connecting rods dip into the oil at the bottom and splash it around the crankcase as needed to lubricate parts inside. In modern vehicle engines, the oil pump takes oil from the oil pan and sends it through the oil filter into oil galleries, from which the oil lubricates the main bearings holding the crankshaft up at the main journals and camshaft bearings operating the valves. In typical modern vehicles, oil pressure-fed from the oil galleries to the main bearings enters holes in the main journals of the crankshaft. From these holes in the main journals, the oil moves through passageways inside the crankshaft to exit holes in the rod journals to lubricate the rod bearings and connecting rods. Some simpler designs relied on these rapidly moving parts to splash and lubricate the contacting surfaces between the piston rings and interior surfaces of the cylinders. However, in modern designs, there are also passageways through the rods which carry oil from the rod bearings to the rod-piston connections and lubricate the contacting surfaces between the piston rings and interior surfaces of the cylinders. This oil film also serves as a seal between the piston rings and cylinder walls to separate the combustion chamber in the cylinder head from the crankcase.

2.4 Thickener

Thickener is also known as soap. Numerous types of grease thickeners are currently in use, each will give different properties towards the grease. The most common types are simple lithium soaps, lithium complex and polyurea (Gallegos C. *et al.*, 2005). Simple lithium soaps are often used in low-cost general-purpose greases and perform relatively well in most performance categories at moderate temperatures. Complex greases such as lithium complex provide improved performance particularly at higher operating temperatures. A common upper operating temperature limit for simple lithium grease might be 250°F, while that for lithium complex grease might be 350°F. Another thickener type that is becoming more popular is polyurea. Like lithium complex, polyurea has good high-temperature performance as well as high oxidation stability and bleed resistance. Thickener type should be selected based on performance requirements as well as compatibility when considering changing product types. (R.L Marker, 1945) General types of thickener are discussed below:

2.4.1 Calcium Grease. (Atanu Adharvu *et al.*, 2005)

Calcium or lime grease, the first of the modern production greases, is prepared by reacting mineral oil with fats, fatty acids, a small amount of water, and calcium hydroxide (also known as hydrated lime). The water modifies the soap structure to absorb mineral oil. Because of water evaporation, calcium grease is sensitive to elevated temperatures. It dehydrates at temperatures around 79 °C (175 °F) at which its structure collapses, resulting in softening and, eventually, phase separation. Greases with soft consistencies can dehydrate at lower temperatures while greases with firm consistencies can lubricate satisfactorily to temperatures around 93 °C (200 °F). In spite of the temperature limitations, lime grease does not emulsify in water and is excellent at resisting “wash out.” Also, its manufacturing cost is relatively low. Calcium complex grease is prepared by adding the salt calcium acetate. The salt provides the grease with extreme pressure characteristics without using an additive. Dropping points greater than 260 °C (500 °F) can be obtained and the maximum usable temperature increases to approximately 177 °C (350 °F). With the exception of poor pumpability in high-pressure centralized systems, where caking and hardening sometimes occur calcium complex greases have good all-around characteristics that make them desirable multipurpose greases.

2.4.2 Sodium Grease.

Sodium grease was developed for use at higher operating temperatures than the early hydrated calcium greases. Sodium grease can be used at temperatures up to 121 °C (250 °F), but it is soluble in water and readily washes out. Sodium is sometimes mixed with other metal soaps, especially calcium, to improve water resistance. Although it has better adhesive properties than calcium grease, the use of sodium grease is declining due to its lack of versatility. It cannot compete with water-resistant, more heat-resistant multipurpose greases. It is, however, still recommended for certain heavy-duty applications and well-sealed electric motors. (R.L Merker *et al.*, 1945)